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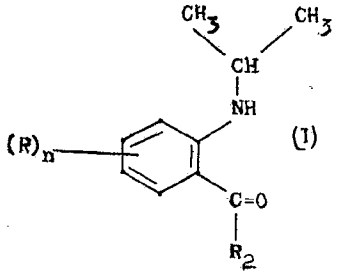
(54) *o*-ISOPROPYLAMINOBENZOPHENONES

(71) We, SANDOZ LTD., of Lichtstrasse 35, Basle, Switzerland, a Swiss Body Corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement: —

This invention relates to quinazolinones and to an improved process for the preparation of o-isopropylaminobenzophenones useful in their production.

The preparation of o-alkylaminobenzophenones has heretofore been accomplished by first tosylating o-aminobenzophenone, alkylating the resulting tosylated intermediate and then removing the protecting tosyl group by hydrolysis. While this process is suitable for preparing o-alkylaminobenzophenones wherein the alkyl moiety is a straight chain alkyl, it is not particularly suitable for the preparation of those compounds wherein the alkyl moiety is a branched chain and the branching occurs on the carbon atom directly attached to the ring nitrogen atom. as there is a significant reduction in the yield of the desired product due to steric hindrance.

In accordance with the present invention there is provided a process whereby o-isopropylaminobenzophenones are obtainable with facility in one step and in excellent yields, which process is characterised by obtaining compounds of formula I,



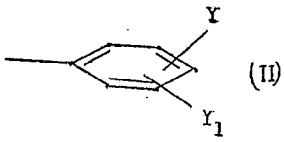
(I)

in which

R signifies a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, a nitro group of a trifluoromethyl group, or an alkylthio radical of 1 to 4 carbon atoms,

n represents 0, 1 or 2, provided that when n is 2 the R's, which may be the same or different, each signify an alkyl or alkoxy radical or a halogen atom, and

R_2 signifies a phenyl radical or a substituted phenyl radical of formula II,



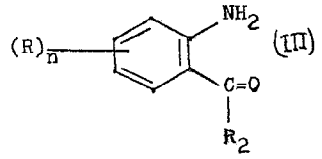
(II)

in which

Y signifies a fluorine, chlorine or bromine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, or a trifluoromethyl group, and

Y_1 signifies a hydrogen atom, a fluorine, chlorine or bromine atom, an alkyl radical of 1 to 4 carbon atoms, or an alkoxy radical of 1 to 4 carbon atoms,

by reacting an o-aminobenzophenone of formula III,



(III)

in which R, n and R_2 are as defined above, with a compound of formula IV,

$(CH_3)_2CHX$ IV

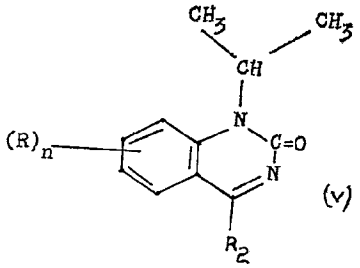
in which X signifies a bromine or iodine atom.

In this process, the compound of formula IV is preferably isopropyl iodide. Desirably, the reaction is carried out in the presence of an inorganic base as acid binding agent, e.g. an alkali metal carbonate such as sodium carbonate, to take up the hydrogen halide liberated during the reaction. If desired, the reaction may be carried out in any suitable inert organic solvent, e.g. dioxane, benzene and toluene. However, the use of a solvent is not necessary since an excess of the isopropyl halide of formula IV can be utilized for this purpose. The reaction is most conveniently effected at elevated temperatures. Desirably, the reaction is carried out at a temperature of from 60° to 100°C. Preferably, the reaction is conducted at the reflux temperature of the system. However, the particular temperature employed is not critical and somewhat lower temperatures or higher temperatures and elevated pressures can be employed.

It should be noted that the reaction proceeds independently of the particular substituents attached to the phenyl rings. Accordingly, while the process of the invention is specifically exemplified with respect to certain substituents attached to either or both of the phenyl rings, it nevertheless can be utilized for the preparation of all of the compounds included within the scope of the invention.

Various of the o-aminobenzophenones (III) employed as starting materials in the process of this invention are known and can be prepared as described in the literature. Such others which may not be specifically described in the literature can be readily prepared from available materials in analogous manner.

The compounds of formula I are useful as intermediates, particularly for the preparation of the corresponding 1-isopropyl-4-aryl-2(1H)-quinazolinones of formula V,



(V)

in which R, n and R_2 are as defined above.

The compounds of formula V may be obtained, for example, by reacting a compound of formula I with a lower alkyl (C_1—C_5) carbamate in the presence of a catalytic amount of a Lewis acid. The reaction is conveniently carried out at elevated temperature, i.e. of at least 140°C, preferably from 160° to 200°C, the preferred Lewis acid being zinc chloride and the preferred carbamate being ethyl carbamate. If desired the reaction may be carried out in the presence of a suitable inert organic solvent. However, the use of a solvent is not necessary since an excess of the carbamate can be used for this purpose.

The compounds of formula V thus produced may be recovered and purified using conventional techniques.

Certain compounds of formula V, namely those wherein R signifies a fluorine, bromine or chlorine atom, n represents 0 or 1 and R_2 is as defined above, have been disclosed in our Application No. 38358/67 (Serial No. 1,195,066). In the latter Application the 1-substituent is an alkyl radical of 1 to 5 carbon atoms generally, or various other radicals, but the present 1-isopropyl compounds in general have particularly good pharmacological properties. The remaining compounds of formula V are disclosed and claimed in our copending Applications No. 5791/71 and No. 5792/71 (Serial Nos. 1248429 and 1248430).

The present invention also provides those compounds of formula I wherein R signifies the above halogen atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, a nitro group or a trifluoromethyl group, or an alkylthio radical of 1 to 4 carbon atoms, n represents 1 or 2, provided that when n is 1, R does not signify a halogen atom, and that when n is 2, the R's which may be the same or different, each signify an alkyl or alkoxy radical or the above halogen atom, and R_2 is as defined above.

The compounds of formula V have pharmacological activity. In particular they have anti-inflammatory activity as indicated by the Carrageen-induced edema test on rats, and are indicated for use as anti-inflammatory agents. Suitable indicated daily dosages range from 40 to 1000 mg, e.g. 150 to 600 mg, preferably given in divided dosages of from 10 to 500 mg, e.g. 37.5 to 300 mg, two to four times a day or in retard form. Compounds of formula V also possess analgesic, antipyretic and anti-bradykinin activity and indicated dosages for analgesic and antipyretic use are similar to those indicated for anti-inflammatory use.

The compounds may be used mixed with a pharmaceutically acceptable carrier, and such other conventional adjuvants as may be desired, and administered orally in such forms as tablets, capsules, elixirs, suspensions or solutions or parenterally in such forms as injectable solutions and suspensions.

A representative formulation is a tablet prepared by conventional tabletting techniques and containing, by weight, 50 parts of a compound of formula V, 2 parts of tragacanth, 39.5 parts of lactose, 5 parts of corn starch, 3 parts of talcum and 0.5 parts of magnesium stearate.

Compounds of particular interest as regards the anti-inflammatory action are those wherein R is methyl or chlorine. One may specifically mention, for example, 7 - methyl - 1 - iso - propyl - 4 - phenyl - 2(1H) - quinazolinone and 6 - methyl - 1 - isopropyl - 4 - phenyl - 2(1H)-quinazolinone.

The following Examples further illustrate the invention.

EXAMPLE 1:

o - Isopropylaminobenzophenone

A mixture of 20 g. of o-aminobenzophenone, 10 g. of sodium carbonate and 50 ml. of isopropyl iodide is refluxed with stirring for 5 days. The excess isopropyl iodide is then evaporated off *in vacuo*, and the resulting residue extracted with 200 ml. of benzene. The benzene extract is then filtered, washed twice with 100 ml. (each) of water, dried over anhydrous sodium sulfate, filtered and evaporated to dryness *in vacuo* to obtain o-isopropylaminobenzophenone as an oil.

EXAMPLE 2:

5-Chloro-2-isopropylaminobenzophenone

A mixture of 10 g. of 5-chloro-2-aminobenzophenone, 5 g. of sodium carbonate and 30 ml. of isopropyl iodide is refluxed with stirring for 2½ days. The excess isopropyl iodide is then evaporated off *in vacuo* and the resulting residue extracted with 200 ml. of benzene. The benzene extract is then filtered, washed twice with 100 ml. (each) of water, dried over anhydrous sodium sulfate, filtered and evaporated to dryness *in vacuo* to obtain 5 - chloro - 2 - isopropylaminobenzophenone as an oil.

EXAMPLE 3:

2-Isopropylamino-4′-methylbenzophenone

A mixture of 5 g. of 2-amino-4′-methylbenzophenone, 5 g. of sodium carbonate and 20 ml. of isopropyl iodide is refluxed with stirring for 5 days. The excess isopropyl iodide is then evaporated off *in vacuo* and the resulting residue extracted with 200 ml of benzene. The benzene extract is then filtered, washed twice with 100 ml (each) of water, dried over anhydrous sodium sulfate, filtered and evaporated to dryness *in vacuo* to obtain 2 - isopropylamino - 4′ - methylbenzophenone as an oil. A procedure differing slightly as regards experimental detail is as follows:

A mixture of 9.5 g of 2-amino-4′-methylbenzophenone, 10 g of sodium carbonate and 30 ml of isopropyl iodide is refluxed for five days, poured onto 200 ml ice water, and extracted three times with 100 ml ethyl acetate. The organic phase is separated, dried over anhydrous sodium sulphate, filtered and evaporated to dryness *in vacuo* to obtain 10 g of reaction mixture as a crude oil which is purified by column chromatography using silica gel with benzene as eluant to obtain a pure yellow oil of 2-isopropylamino-4′-methyl-benzophenone.

EXAMPLE 4:

4,5-Dimethyl-2-isopropylaminobenzophenone

A mixture of 9.5 g. of 2-amino-4,5-dimethylbenzophenone, 5 g. of sodium carbonate and 30 ml. of isopropyl iodide is refluxed with stirring for 20 hours. The excess isopropyl iodide is then evaporated off *in vacuo* and the resulting residue extracted with 200 ml. of benzene. The benzene extract is then filtered, washed twice with 100 ml. (each) of water, dried over anhydrous sodium sulfate, filtered and evaporated to dryness *in vacuo* to obtain 4,5 - dimethyl - 2 - isopropylamino - benzophenone as an oil which is further purified chromatographically on an aluminum oxide column.

A procedure differing slightly as regards experimental detail is as follows:

A mixture of 9.5 g. of 4,5-dimethyl-2-aminobenzophenone, 10 g. of sodium carbonate and 30 ml. of isopropyl iodide is refluxed for 20 hours, poured onto 200 ml. ice water, and extracted three times each with 100 ml. of ethyl acetate. The organic phase is separated, dried over anhydrous sodium sulphate, filtered, and the filtrate evaporated to dryness *in vacuo* to obtain 10 g. of reaction mixture as a crude oil. The oil is purified by column chromatography using silica gel with benzene as eluant to obtain a pure yellow oil of 4,5-dimethyl-2-isopropylaminobenzophenone.

EXAMPLE 5:

2 - Isopropylamino - 5 - trifluoromethyl - benzophenone

A mixture of 25 g. of 2-amino-5-trifluoromethylbenzophenone, 15 g. of sodium carbonate and 100 ml. of isopropyl iodide is refluxed with stirring for 4 days. The excess isopropyl iodide is then evaporated off *in vacuo* and the resulting residue extracted with 200 ml. of benzene. The benzene extract is then filtered, washed twice with 100 ml. (each) of water, dried over anhydrous sodium sulfate, filtered and evaporated to dryness *in vacuo* to obtain 2 - isopropylamino - 5 - trifluoromethyl - benzophenone as an oil. The oil is crystallized from cold ethanol to obtain product; m.p. 68° —70°C.

EXAMPLE 6:

5-Methyl-2-isopropylaminobenzophenone

Proceeding as in Example 4 (second procedure) and using equivalent amounts, 5-methyl-2-aminobenzophenone in admixture with sodium carbonate and isopropyl iodide is refluxed for 4 days. Completing the procedure as in Example 4 (second procedure), there is obtained a pure yellow oil of 5-methyl-2-isopropylaminobenzophenone.

EXAMPLE 7:

4-chloro-2-isopropylaminobenzophenone

Employing equivalent amounts and proceed-

ing as in Example 4 (second procedure), except that reflux is conducted over a period of four days, 4 - chloro - 2 - aminobenzophenone is reacted to obtain from the column chromatography a purified yellow oil of 4-chloro-2-isopropylaminobenzophenone.

EXAMPLE 8:

4,5 - dimethoxy - 2 - isopropylaminobenzophenone

Employing equivalent amounts 4,5-dimethoxy-2-aminobenzophenone is reacted according to the procedure of Example 4 (second procedure) except that the reflux is conducted for a period of 2½ days. The product obtained from the column chromatography is a purified yellow oil of 4,5-dimethoxy-2-isopropylaminobenzophenone.

EXAMPLE 9:

4-methyl-2-isopropylaminobenzophenone

A mixture of 7 g. of 4-methyl-2-aminobenzophenone, 6.35 g. of sodium carbonate and 18.8 ml. of 2-iodopropane is stirred and refluxed for 3 days. The cooled reaction mixture is then diluted with 200 ml. of benzene and washed twice with water and twice with brine. The organic phase is separated, dried over anhydrous sodium sulfate and concentrated *in vacuo* to remove substantially all of the benzene. The resulting yellow oil is dissolved in about 10 ml. of methylene chloride and subjected to column chromatography employing alumina (about 400 g.) and methylene chloride as eluant to give a first fraction which on concentration *in vacuo* to remove methylene chloride produced a yellow oil of 4-methyl-2-isopropylaminobenzophenone.

EXAMPLE 10:

4 - methoxy - 2 - isopropylaminobenzophenone

A mixture of 9 g. of 2-amino-4-methoxybenzophenone, 15 g. of anhydrous potassium carbonate and 40 ml. of 2-iodopropane is refluxed for 4 days. The cooled reaction mixture is then diluted with 200 ml. of benzene and washed twice with water and twice with brine. The organic phase is separated, dried over anhydrous sodium sulfate and concentrated *in vacuo* to remove substantially all of the benzene. The resulting oil is dissolved in about 10 ml. of methylene chloride and subjected to column chromatography employing alumina (about 400 g.) and methylene chloride as eluant to give a first fraction which on concentration *in vacuo* to remove methylene chloride produced an oil of 4-methoxy-2-isopropylaminobenzophenone.

EXAMPLE 11:

3,5 - dimethyl - 2 - isopropylaminobenzophenone

A mixture of 14.6 g. of 2-amino-3,5-dimethylbenzophenone (prepared by reactions known from Sternbach et al., J. Org. Chem. 27, 3781 (1962)), 15 g. of anhydrous potassium carbonate and 36 ml. of 2-iodopropane is refluxed for 4 days and then heated for 24 hours in a sealed vessel at a temperature of 160°C. The cooled reaction mixture is then diluted with 200 ml. of benzene and washed twice with water and twice with brine. The organic phase is separated, dried over anhydrous sodium sulfate and concentrated *in vacuo* to remove substantially all of the benzene. The resulting oil is dissolved in about 10 ml. of methylene chloride and subjected to column chromatography employing alumina (about 400 g.) and methylene chloride as eluant to give a first fraction which on concentration *in vacuo* to remove methylene chloride produced an oil of 3,5 - dimethyl - 2 - isopropylaminobenzophenone.

EXAMPLE 12:

4,6 - dimethyl - 2 - isopropylaminobenzophenone

A mixture of 5 g. of 2-amino-4,6-dimethylbenzophenone (prepared by reactions known from E. Ritchie, J. Proc. Roy. Soc. N.S. Wales 80, 33 (1946) and Chem. Abstracts 41, 3094(c) (1947)), 5 g. of anhydrous potassium carbonate and 20 ml. of 2-iodopropane is refluxed for 30 hours. The cooled reaction mixture is then diluted with 200 ml. of benzene and washed twice with water and twice with brine. The organic phase is separated, dried over anhydrous sodium sulfate and concentrated *in vacuo* to remove substantially all of the benzene. The resulting oil is dissolved in about 10 ml. of methylene chloride. The resulting solution is diluted with pentane and concentrated in vacuo to crystallize 4,6-dimethyl - 2 isopropylaminobenzophenone; m.p. 87°—88°C.

EXAMPLE 13:

2-isopropylamino-6-methylbenzophenone

A mixture of 1.4 g. of 2-amino-6-methylbenzophenone, 2 g of anhydrous potassium carbonate and 20 ml of 2-iodopropane is refluxed for 130 hours. The cooled reaction mixture is then diluted with 200 ml of benzene and washed twice with water and twice with brine. The organic phase is separated, dried over anhydrous sodium sulfate and concentrated *in vacuo* to remove substantially all of the benzene. The resulting oil is dissolved in about 10 ml of methylene chloride and subjected to column chromatography employing alumina (about 50 g) and methylene chloride as eluant to give a first fraction which on concentration *in vacuo* to remove methylene chloride produced an oil of 2-isopropylamino-6-methylbenzophenone.

EXAMPLE 14:

Employing the reaction of Example 9 and the appropriate 2-amino-benzophenone starting materials in approximately equivalent amounts, there are obtained:

4 - ethyl - 2 - isopropylaminobenzophenone
4,5 - dichloro - 2 - isopropylaminobenzophenone
4 - methylthio - 2 - isopropylaminobenzophenone
5-ethyl-2-isopropylaminobenzophenone
4 - methyl - 2 - isopropylamino - 4' - methylbenzophenone
4 - methyl - 2 - isopropylamino - 4' - methoxybenzophenone

EXAMPLE 15:

5 - methylthio - 2 - isopropylaminobenzophenone

Proceeding as in Example 4 (second procedure), above, 5-methylthio-2-aminobenzophenone is reacted to obtain after purification a yellow oil of 5-methylthio-2-isopropylaminobenzophenone.

EXAMPLE 16:

4 - chloro - 2 - isopropylamino - 5 - methylbenzophenone

Step A: 2 - amino - 4 - chloro - 5 - methylbenzophenone

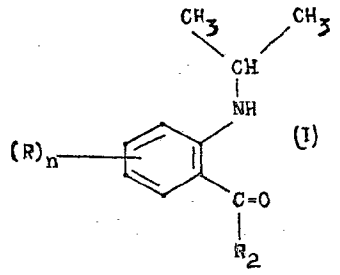
To 142 g. of benzoyl chloride is added a total of 57 g. of 3-chloro-4-methylaniline in small portions over a period of 1/2 hour at temperature of 110°C. The resulting mixture is heated to a temperature of 180°C. 140 g. of zinc chloride added in divided portions over 1 hour and heating then continued at temperature of 225—230°C. for 1 1/2 hours. The resulting mixture is cooled to a temperature of 120—130°C. and there is added a mixture of 150 ml. of acetic acid, 100 ml. of water and 150 ml. of concentrated sulfuric acid. The resulting mixture is then refluxed for 3 hours, poured onto 2 liters of ice and water and extracted 3 times each with 300 ml. of methylene chloride. The organic layers are combined, dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo*. The resulting oily residue is distributed between a liquid system composed of 500 ml. of 2N sodium hydroxide and 300 ml. of methylene chloride and the organic phase washed with water, then with brine, dried, filtered and evaporated *in vacuo* to obtain a crude material which is purified by crystallization from diethyl ether/pentane (1:2) to obtain 2 - amino - 4 - chloro - 5 - methylene - benzophenone, m.p. 95—96°C.

Step B: 4 - chloro - 2 - isopropylamino - 5 - methylbenzophenone

A mixture of 22 g. of 2-amino-4-chloro-5-methylbenzophenone obtained from Step A, above, 20 g. of sodium carbonate and 60 ml. of 2-iodopropane is refluxed for 120 hours. The cooled reaction mixture is then diluted with 300 ml. of methylene chloride and extracted twice each with 100 ml. of water. The organic phase is separated, dried over anhydrous sodium sulfate, filtered and evaporated *in vacuo* to remove solvent. The resulting oil is dissolved in a small amount of methylene chloride and subjected to column chromatography employing alumina and methylene chloride as eluant to give a first fraction which on concentration *in vacuo* to remove methylene chloride produced an orange oil of 4 - chloro - 2 - isopropylamino - 5 - methylbenzophenone.

WHAT WE CLAIM IS:—

1. A process for obtaining compounds of formula I,



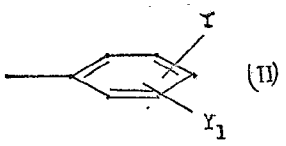
(I)

in which

3 signifies a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, a nitro group or a trifluoromethyl group, or an alkylthio radical of 1 to 4 carbon atoms,

n represents 0, 1 or 2, provided that when n is 2 the R's, which may be the same or different, each signify an alkyl or alkoxy radical or a halogen atom, and

R_2 signifies a phenyl radical or a substituted phenyl radical of formula II,



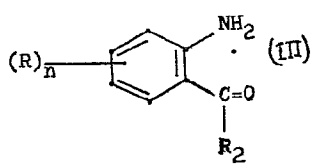
(II)

in which

Y signifies a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, or a trifluoromethyl group, and

Y_1 signifies a hydrogen atom, a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, or an alkoxy radical of 1 to 4 carbon atoms,

characterised by reacting an o-aminobenzophenone of formula III,



(III)

in which R, n and R_2 are as defined above, with a compound of formula IV,

$$(CH_3)_2CHX \qquad IV$$

in which X signifies a bromine or iodine atom.

2. A process according to Claim 1, wherein there is used isopropyl iodide as the compound of formula IV.

3. A process according to Claim 1 or 2, wherein the reaction is carried out in the presence of an inorganic base to take up the hydrogen halide liberated in the reaction and at a temperature of from 60° to 100°C.

4. A process according to any one of Claims 1 to 3, for the production of a compound of formula I, depicted in Claim 1, wherein R signifies a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms or a nitro or trifluoromethyl group, n represents 0, 1 or 2 and R_2 is as defined in Claim 1, provided that when n is 2 the R's are the same and either an alkyl or alkoxy radical.

5. A process for the production of a compound of formula I, stated in Claim 1, substantially as hereinbefore described in any one of Examples 1 to 16.

6. A compound of formula I, as defined in Claim 1, whenever obtained by a process claimed in any one of Claims 1 to 3 and 5.

7. A compound of formula I, depicted in Claim 1, in which R signifies a fluorine, a chlorine or bromine atom, an alkyl, an alkoxy radical of 1 to 4 carbon atoms, or a nitro or trifluoromethyl group, n represents 0, 1 or 2, and R_2 is as defined in Claim 1, provided that, when n is 2, the R's are the same and signify an alkyl or alkoxy radical, whenever produced by a process according to Claim 4.

8. A compound of formula I, as depicted in Claim 1, wherein R signifies a fluorine, bromine or chlorine atom, an alkyl radical of 1 to 4 carbon atoms, an alkoxy radical of 1 to 4 carbon atoms, a nitro group or a trifluoromethyl group, or an alkylthio radical of 1 to 4 carbon atoms, n represents 1 or 2, provided that when n is 1, R does not signify said halogen atom, and that when n is 2, the R's, which may be the same or different, each signify an alkyl or alkoxy radical or said halogen atom, and R_2 is as defined in Claim 1.

9. A compound of formula I, depicted in Claim 1, in which R signifies a fluorine, chlorine or bromine atom, an alkyl or alkoxy radical of 1 to 4 carbon atoms, or a nitro or trifluoromethyl group, n represents 1 or 2 and R_2 is as defined in Claim 1, provided that when n is 1, R does not signify a halogen atom and that when n is 2, the R's are the same and signify an alkyl or alkoxy radical.

10. 4,5 - dimethyl - 2 - isopropylamino - benzophenone.

11. 2 - isopropylamino - 5 - trifluoro - methylbenzophenone.

12. 5 - methyl - 2 - isopropylaminobenzo - phenone.

13. 4,5 - dimethoxy - 2 - isopropylamino - benzophenone.

14. 4 - methyl - 2 - isopropylaminobenzo - phenone.

15. 4 - methoxy - 2 - isopropylaminobenzo - phenone.

16. 3,5 - dimethyl - 2 - isopropylamino - benzophenone.

17. 4,6 - dimethyl - 2 - isopropylamino - benzophenone.

18. 2 - isopropylamino - 6 - methylbenzo - phenone.

19. 4 - ethyl - 2 - isopropylaminobenzo - phenone.

20. 4,5 - dichloro - 2 - isopropylamino - benzophenone.

21. 4 - methylthio - 2 - isopropylamino - benzophenone.

22. 5 - ethyl - 2 - isopropylaminobenzo - phenone.

23. 4 - methyl - 2 - isopropylamino - 4' - methylbenzophenone.

24. 4 - methyl - 2 - isopropylamino - 4' - methoxybenzophenone.

25. 5 - methylthio - 2 - isopropylamino benzophenone.

26. 4 - chloro - 2 - isoproplamino - 5 - methylbenzophenone.

For the Applicants:—
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